

# PATENT ABSTRACTS OF JAPAN

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(71)Applicant : SUMITOMO METAL IND LTD

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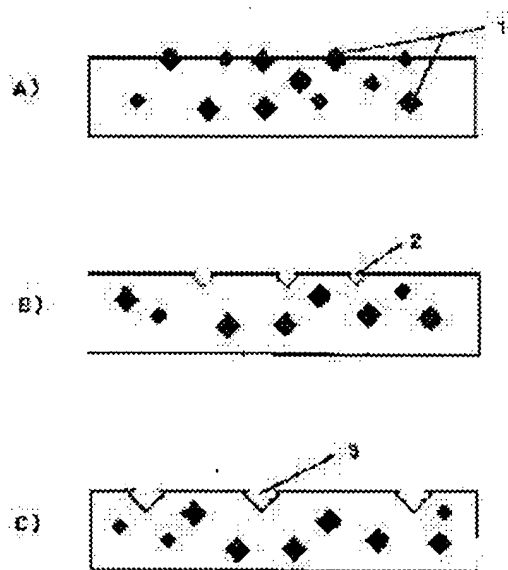
(72)Inventor : KOIKE YASUO

## (54) EVALUATION OF SEMICONDUCTOR SUBSTRATE

### (57)Abstract:

**PROBLEM TO BE SOLVED:** To enable observation on the entire surface of substrate to a predetermined depth in a wide range and with high sensitivity, and evaluation from the observation, by increasing the size of pits formed by etching and exposing the surface of the substrate, and measuring a pit density by a laser foreign matter detecting device.

**SOLUTION:** Oxygen precipitates 1 are cleaned with aqueous solution having HF concentration of 20 to 50% for about 30 minutes, thus the oxygen precipitates 1 exposed on the surface of a wafer are dissolved. The HF cleaning forms pits 2 on the wafer surface. The density of pits 2 is measured by the laser foreign matter detecting device. The current minimum detection size of the laser foreign matter detecting device is about 110 nm. If there is a pit of a size smaller than this size, all the pits cannot be measured. Accordingly, cleaning is performed 5 to 10 cycles (10 minutes/cycle) by using alkali cleaning liquid composed of  $\text{NH}_4\text{OH}/\text{H}_2\text{O}_2/\text{H}_2\text{O}$ , attaining a comparatively large amount of silicon etching. The cleaning greatly changes the width of pits 3. Next, the laser foreign matter detecting device may detect oxygen precipitates of about several 10 nm, in defective distribution on the entire wafer surface.



## LEGAL STATUS

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## DETAILED DESCRIPTION

### [Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the evaluation approach for being manufactured using a CZ process, evaluating the crystal defect in the semi-conductor substrate which performed various heat treatments, and manufacturing a quality semi-conductor substrate with few crystal defects. By removing an oxide for the consistency of the minute oxygen sludge which exists in the bulk near the pole front face of a semi-conductor substrate by HF washing, exposing a pit, and expanding pit size by alkali cleaning further It is related with the evaluation approach of the semi-conductor substrate whose observation and evaluation to high sensitivity the whole substrate surface enabled wide range with laser foreign matter detection equipment.

[0002]

[Description of the Prior Art] CZ (CHOKURARU skiing) -- although the integrity of the crystal defect near the front face of the semi-conductor substrate manufactured using law improves partly by performing high temperature processing (1000 degrees C - 1200 degrees C), it is not perfect and is imperfect compared with the epitaxial wafer which carried out epitaxial membrane formation on a front face.

[0003] In order to produce a semiconductor device, to a semi-conductor substrate, it is desirable for a defect not to exist in a device property about the field of 10-micrometer depth extent from the field which does effect, and a front face, but unless an epitaxial wafer is generally used, the present condition is that it is difficult to make said field defect-free with the semi-conductor substrate manufactured by the CZ process.

[0004] However, since the cost of a substrate costs dearly by carrying out epitaxial membrane formation, as a substrate for MOS mold LSI processes, the substrate which performed high temperature processing is used widely. Moreover, in a device process, even if it does not perform high temperature processing beforehand from 500 degrees C - 1200 degrees C heat treatment being planned, although the integrity of the crystal defect near the front face improves a little by device process heat treatment, it is not perfect like the substrate which performed high temperature processing.

[0005] although the defect-free layer called DZ (Deneuded Zone) layer near the front face of a substrate is deeply formed so that it is heat-treating about the class of crystal defect which exists near the front face and the temperature is an elevated temperature -- parenchyma -- a 1 hole type Grown-in defect and 2 -- the minute oxygen sludge exists and it is known well that it is not a defect-free layer.

[0006]

[Problem(s) to be Solved by the Invention] as the approach of evaluating the consistency of the crystal defect which exists near the front face of these semi-conductors substrate -- a selective etching method, the IR tomography method, and OPP -- it is measured by the COP appraisal method about law or Grown-in. if it explains in full detail -- a selective etching method -- mainly -- as a crystal defect appraisal method -- current -- it uses widely -- having -- \*\*\*\* -- M.Dash The ET method and Wright Although the ET method is raised, the detection sensitivity to a minute defect is low, it is very difficult

at the time of observation by the optical microscope after ET to judge it as a defect, and it is impossible to detect a Grown-in defect.

[0007] As an ET method for detecting a Grown-in defect, it is Secco. Although the ET method is raised, the defect detected by this approach is called FPD (Flow Pattern Defect) and SPD (Secco Pit Defect) and it is reported by the Japan Society of Applied Physics etc., it is difficult for particle to adhere at the time of ET and to detect the consistency of a pit correctly with an optical microscope.

[0008] the IR tomography method and OPP -- when dozens of hours tend to be required for evaluating the whole surface of an evaluation substrate broadly and it is going to evaluate only the depth of a certain field from a front face, law can perform only evaluation in the large range of 5 micrometers and 10-micrometer step on the problem of the diameter of laser, but is unsuitable to evaluation of a narrow specific region. Moreover, the detection sensitivity to a minute defect is difficult for the defect density which it may be greatly influenced by the condition on the rear face of front of an evaluation substrate, and is defended stoutly in that case to show an unusually high value, and to distinguish the consistency of only a defect. Moreover, although this appraisal method is suitable for evaluating the defective information near the front face of the substrate which performed evaluation and heat treatment of the substrate which is not heat-treating of a Grown-in defect on internal in the comparatively large range, evaluation in the narrow range of 1-2 micrometers cannot be performed.

[0009] Although a COP appraisal method can evaluate the consistency of the Grown-in defect of a substrate to the large area of the whole substrate surface by repeating alkali cleaning and performing it and it is the approach of measuring defect density using a laser \*\*\*\* machine Since COP is embedded at an oxide film and the irregularity of a defect becomes small, when various evaluation heat treatments are performed, Since the defective detection by the laser \*\*\*\* machine is already an oxide, concerning a minute oxygen sludge in becoming difficult \*\*\*\*, the irregularity of a defect is small and defective detection is difficult only at alkali cleaning.

[0010] in short -- the IR tomography method and OPP -- it was very difficult for a measurement field to be narrow and to evaluate the large area of the whole surface of a semi-conductor substrate by law to high sensitivity about a minute oxygen sludge. Moreover, since a measurement result in the large fields spaced [ 5 micrometers and ] at 10 micrometers was brought on the problem of the diameter of laser when it is going to acquire the information only on the field which is near the front face, measurement at a fine step was difficult.

[0011] This invention relates to the semi-conductor substrate which performed what performed high temperature processing to the semi-conductor substrate manufactured using the CZ process, heat treatment (500 degrees C - 1200 degrees C) supposing a device process, etc. It is what is proposed in view of there having been no approach of measuring the minute oxygen sludge which exists near the front face of a semi-conductor substrate simple [ to high sensitivity ] broadly [ the whole substrate surface ] conventionally. It aims at offer of the evaluation approach of a semi-conductor substrate whose observation and evaluation of the depth predetermined to high sensitivity with the wide range and whole substrate surface were enabled with common laser foreign matter detection equipment.

[0012]

[Means for Solving the Problem] The result to which the artificer examined variously the consistency of the minute oxygen sludge which exists in the bulk near the pole front face of the heat-treated semi-conductor substrate for the purpose of the wide range and evaluation approach observable to high sensitivity, After the water solution of 20 - 50% of HF concentration performing HF washing, removing oxide and making it a concave pit configuration, by performing alkali cleaning and etching a silicon substrate It was made to change to pit size detectable [ with laser foreign matter detection equipment ], the knowledge of the ability to evaluate broadly the minute oxygen sludge which exists all over a substrate to high sensitivity was carried out, and this invention was completed.

[0013] Namely, this invention receives the semi-conductor substrate which performed necessary heat treatment. After removing the thermal oxidation film formed of this heat treatment and performing mirror polishing from a front face to an evaluation schedule field, remove the oxidation sludge on the front face of a substrate by HF washing, and a pit is exposed. It is the evaluation approach of the semi-

conductor substrate which expands the size of the pit which etched the substrate front face and was furthermore exposed by alkali cleaning, and measures a pit consistency with laser foreign matter detection equipment.

[0014]

[Embodiment of the Invention] The evaluation approach by this invention consists of processes shown in drawing 1.

Process 1: Give a semi-conductor substrate for crystal defect evaluation (for example, heat treatment of 500 degrees C - 1200 degree-C \*\*). Process 2: Process 3 which removes the thermal oxidation film formed of heat treatment: In order to measure the minute oxygen sludge consistency which exists near the front face of a substrate Process 4 which performs mirror polishing from a front face to the field (- 50 micrometers) of an aim: By HF washing Process 5 which dissolves the sludge exposed to the front face of a semi-conductor substrate: Remove the particle which adhered at the time of HF washing. Process 6: Process 7 to which carry out alkali cleaning and the concave minute pit dissolved by HF washing is made to expand: A pit consistency is measured using laser foreign matter test equipment.

[0015] It is aimed at what performed elevated-temperature heat treatment and IG processing to the wafer by which mirror plane processing was pulled up and carried out by the CZ process in this invention as a semi-conductor substrate which is going to measure a crystal defect consistency, and the thing which performed crystal defect evaluation heat treatment (500 degrees C - 1200 degrees C) supposing a device process to the thing of a condition [ that wafer processing is carried out with them ].

[0016] In order for most semi-conductor substrates which receive the above-mentioned heat treatment to heat-treat in the gas ambient atmosphere of both an oxidizing quality and a non-oxidizing quality (500 degrees C - 1200 degrees C), the thermal oxidation film is formed. In order to remove the oxide film, after performing HF washing and removing the thermal oxidation film, mirror polishing is performed by Fukashi who performs measurement.

[0017] At the polish time, as shown in drawing 2 A, the oxygen sludge 1 exposed to the substrate front face exists. Next, HF washing is performed. HF washing is washing about 30 minutes in the water solution whose HF concentration's is 20 - 50%, and the oxygen sludge 1 exposed to a wafer front face is dissolved. Then, although the particle which adhered at the time of HF washing is removed, alkali-cleaning liquid with few [ specifically ] amounts of etching of silicon than alkali cleaning adopted by this invention washes.

[0018] Of the dissolution of HF washing, the pit 2 shown in drawing 2 B is formed in a wafer front face. Although laser foreign matter test equipment is used for this formed pit next and it measures a consistency, since the minimum-limit-of-detection size of the present laser foreign matter test equipment is about 110nm, when pit size smaller than this exists, it can measure no pits.

[0019] Then, using the comparatively large alkali-cleaning liquid of the amount of silicon etching which consists of  $\text{NH}_4\text{OH}/\text{H}_2\text{O}_2/\text{H}_2\text{O}$ , in order to expand pit size to magnitude measurable [ with laser foreign matter test equipment ], as shown in drawing 2 C, the width of face of a pit 3 changes a lot by performing 5 - 10 cycle washing in 10 minutes/a cycle.

[0020] Next, it is possible for detection of an about dozens of nm very small oxygen sludge to also perform defective distribution of the whole wafer surface to high sensitivity by measuring using laser foreign matter test equipment.

[0021]

[Example]

As an example 1 evaluation substrate, the 6 inch silicon p (100) wafer which was able to be pulled up by the CZ process was used. An initial oxygen density is  $14 \times 10^{17}$  atoms/cm<sup>3</sup>, and specific resistance is 5 ohm-cm. The heat treatment wafer into which the oxygen sludge which performed heat treatment of 2 hours to this sample in the non-oxidizing quality gas ambient atmosphere by 800 degrees C, was made to form a precipitation-of-oxygen nucleus into bulk, and performed and formed heat treatment of 16 hours by the oxidizing gas ambient atmosphere at 1000 degrees C was grown up was produced.

[0022] In order to expose an oxygen sludge on this wafer front face certainly, after removing the thermal oxidation film, 20 micrometers of mirror polishing were given. First, when the etch pit consistency

which performs selective etching (Wright Etch 1min) to the wafer substrate of the condition, and exists in a substrate front face was measured, it was 75-/cm<sup>2</sup>. This is as a result of [ in the etching method ] oxygen sludge densitometry.

[0023] As the pit consistency in the condition (process 3) that the oxygen sludge was exposed to the substrate front face with the sample lot which performed the same heat treatment as the sample which measured by the etching method previously, and 20-micrometer polish was shown in drawing 3 A as a measurement map of laser foreign matter test equipment (Tencor Surfscan), it was 5-/cm<sup>2</sup>.

[0024] Next, they were the drawing 3 B profit and 90-/cm<sup>2</sup> considering the pit consistency after washing and the alkali-cleaning liquid of NH<sub>4</sub> OH:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>O=1:5:20 washing in the water solution of 30% of HF concentration for 10 minutes for 30 minutes and dissolving an oxygen sludge (process 5) as a measurement map of laser foreign matter test equipment.

[0025] As the pit consistency after the alkali-cleaning liquid of NH<sub>4</sub> OH:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>O=1:1:5 performs 5 cycle washing in 10 minutes/a cycle (process 7) was shown in drawing 3 C as a measurement map of laser foreign matter test equipment, it was 188-/cm<sup>2</sup>.

[0026] Compared with the process 5, i.e., the pit consistency after particle removal is completed after HF washing, it is twice [ about ] the pit consistency of this in the time of approach completion of a process 7, i.e., this invention, so that clearly [ drawing 3 ]. Moreover, even if compared with a previous selective etching method, the twice [ about ] as many consistency as this is detectable.

[0027] Next, the pit size in the condition of the above-mentioned process 5 of a sample and a process 7 was measured by AFM. A measurement result is shown in drawing 4 . At the process 7, width of face of 120-250nm, the depth 50-120, and width of face are changing to twice [ about ] as many magnitude as this to being width of face of 50-120nm, and a depth of 30-80nm at a process 5. This means that the dozens of nm minute oxygen sludge which was not able to be detected with laser foreign matter detection equipment became detectable at the process 7 in the above-mentioned process 5.

[0028] As an example 2 evaluation substrate, the 6 inches silicon p (100) wafer which was able to be pulled up by the CZ process was used. An initial oxygen density is 15x10<sup>17</sup> atoms/cm<sup>3</sup>, and specific resistance is 10 ohm-cm. Elevated-temperature heat treatment (an oxidizing quality and non-oxidizing quality gas ambient atmosphere) for forming DZ layer in this sample like the following heat treatment conditions, heat treatment (non-oxidizing gas ambient atmosphere) which forms IG (precipitation-of-oxygen nucleus) in the interior in bulk, and heat treatment (oxidizing gas ambient atmosphere) for growing up that precipitation-of-oxygen nucleus were performed, and the evaluation substrate was produced.

[0029] evaluation substrate A DZ layer 10 micrometers Oxygen sludge consistency 5x10<sup>5</sup>-/cm<sup>2</sup> heat-treatment conditions 1100 degree-Cx1hr->800 degree-Cx3hr->1000 degree-Cx16hrB DZ layer 20 micrometers Oxygen sludge consistency 4x10<sup>5</sup>-/cm<sup>2</sup> heat-treatment conditions 1150 degree-Cx1hr->800 degree-Cx3.5hr->1000 degree-Cx16hrC DZ layer 30 micrometers Enzyme sludge consistency 6x10<sup>5</sup>-/cm<sup>2</sup> heat-treatment conditions 1200 degree-Cx1hr->800 degree-Cx4hr->1000 degree-Cx16hr

[0030] In addition, DZ layer and the oxygen sludge consistency of the above-mentioned evaluation substrate are as a result of [ from the cross section of the substrate by the optical microscope after performing selective etching (Wright Etch 5 minutes) ] measurement, and are the sample of the same lot as the silicon wafer which has in evaluation by this invention and is.

[0031] Since the thermal oxidation film is formed in the evaluation substrate heat-treated and prepared, After the water solution of 10% of HF concentration washing and removing the thermal oxidation film, in order to perform defective density distribution evaluation in DZ layer, In mirror polishing, it ground to 0, 2, 4 and 6 of a front face, and 10 or 30-micrometer depth, and in order to dissolve the oxygen sludge which has projected on the substrate front face, the water solution of 30% of HF concentration washed.

[0032] Furthermore, in order to enlarge a pit configuration, after carrying out five cycles in 10 minutes/a cycle with the alkali-cleaning liquid of NH<sub>4</sub> OH:H<sub>2</sub>O<sub>2</sub>:H<sub>2</sub>O=1:1:5, the defective number was measured with laser foreign matter test equipment. The measurement result is shown in drawing 5 (B). A measurement consistency is a consistency which carried out the volume conversion of the consistency